

FIG. 1A

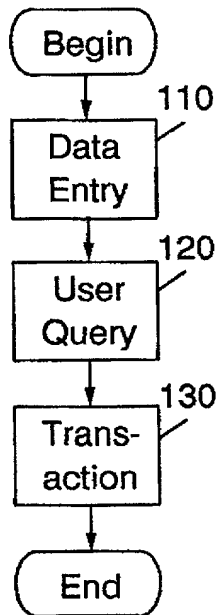


FIG. 1B

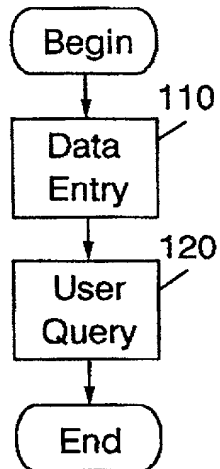


FIG. 1C

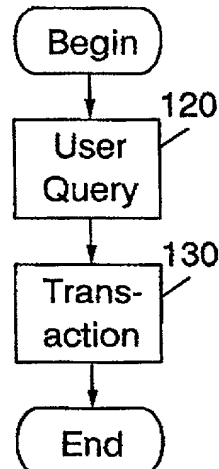


FIG. 1D

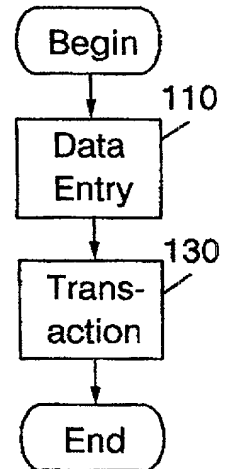


FIG. 1E

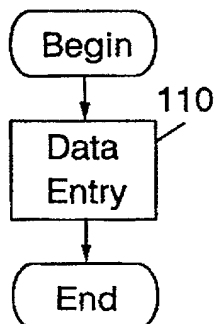


FIG. 1F

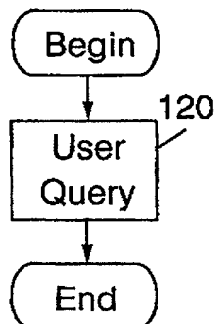


FIG. 1G

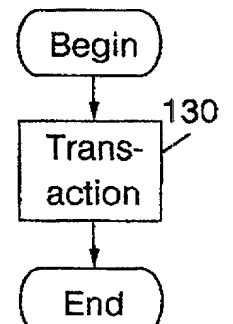


FIG. 2

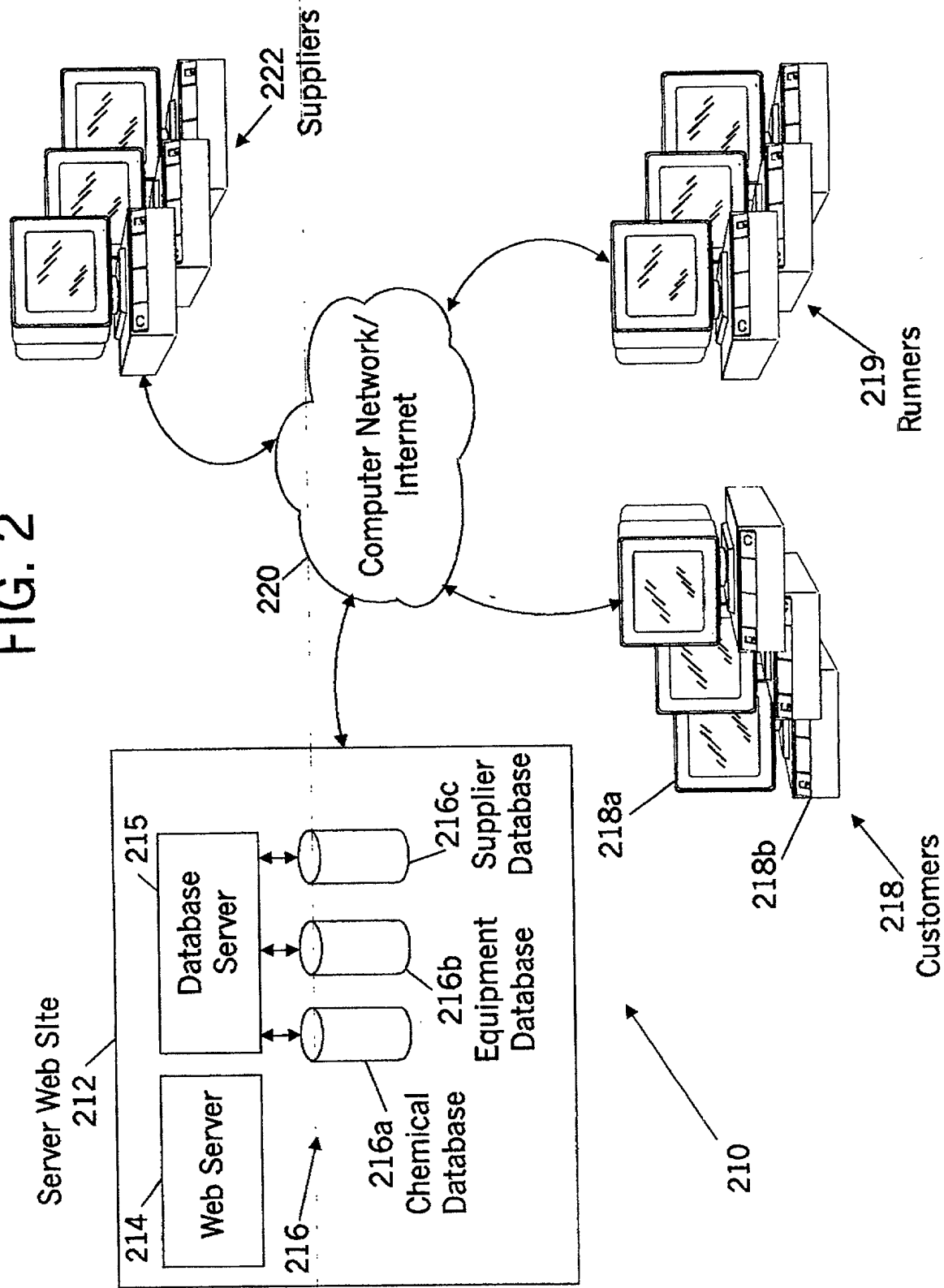
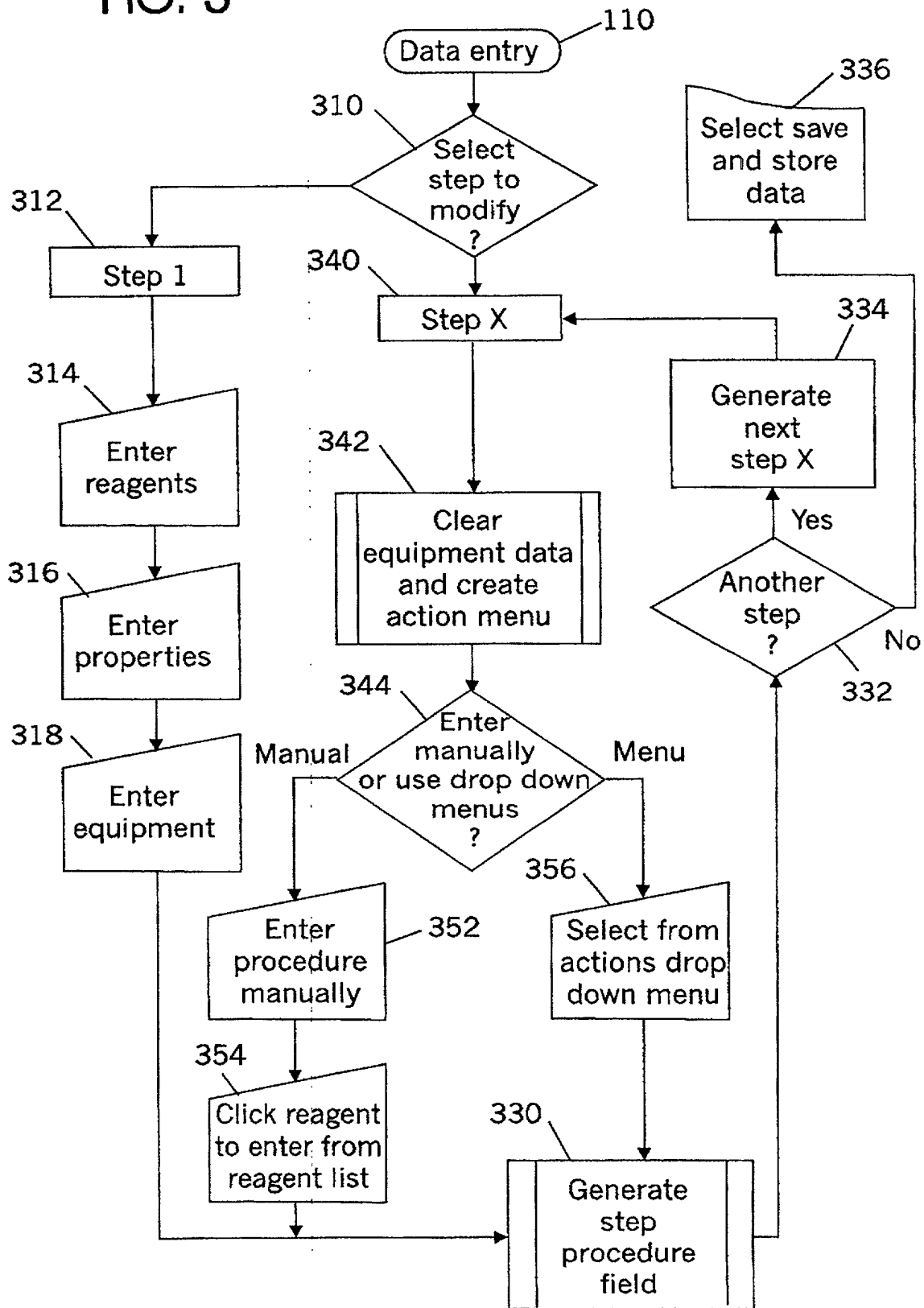


FIG. 3



Synthemix Protocol Manager ver. 0.90b

Name: CAS: Formula: Weight:

Reagents:

	CAS	Name	Weight	Amount (g)
1	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>
2	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>
3	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>
4	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>
5	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>
6	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>
7	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>
8	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>
9	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>
10	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>

Starting Flask:

Equipped with:

<input type="text"/>
<input type="text"/>
<input type="text"/>
<input type="text"/>
<input type="text"/>
<input type="text"/>
<input type="text"/>
<input type="text"/>
<input type="text"/>
<input type="text"/>

Procedure

Step 1

Reaction Type/Keywords (Separated by semicolon)

FIG. 4

Synthematrix Protocol Manager ver. 0.90b

Name: _____ CAS: _____ Formula: _____ Weight: _____

Reagents: ☐ Enter procedure by hand

Search Results

CAS	Chemical Name
71-43-2	Benzene
104-86-9	Benzenemethanamine, 4-chloro-
105-13-5	Benzenemethanol, 4-methoxy-
105-05-5	Benzene, 1,4-diethyl-
118-69-4	Benzene, 1,3-dichloro-2-methyl-
121-14-2	Benzene, 1-methyl-2,4-dinitro-
70-34-8	Benzene, 1-fluoro-2,4-dinitro-
93-05-0	1,4-Benzenediamine, N,N-diethyl-
98-10-2	Benzenesulfonamide
104-51-8	Benzene, butyl-
827-52-1	Benzene, cyclohexyl-
14321-27-8	Benzenemethanamine, N-ethyl-

Procedure

Step 2

Qty		Qty	
Qty	<input type="text"/>	Qty	<input type="text"/>
Qty	<input type="text"/>	Qty	<input type="text"/>
Qty	<input type="text"/>	Qty	<input type="text"/>
Qty	<input type="text"/>	Qty	<input type="text"/>
Qty	<input type="text"/>	Qty	<input type="text"/>

FIG. 5

Synthematrix Protocol Manager ver. 0.90b

Name:

Reagents

	CAS	Name	Weight	Formula	Density	BP	FP	MP	Vapor Pressure	Comments	Beilstein	Other Names
1	101-01-1											
2												
3												
4												
5												
6												
7												
8												
9												
10												

Procedure

Previous:

Save

Cancel

Exit

Hand:

Add To Procedure

New

Save

Update

Qty:

Qty:

Qty:

Qty:

Qty:

Qty:

FIG. 6

Synthematrix Protocol Manager ver. 0.90b

Name: CAS: Formula: Weight:

Reagents: ☐ Enter procedure by hand

	CAS	Name	Weight	Amount (g)	
1	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	Action: <input type="text"/>
2	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	Qualifier: <input type="text"/>
3	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	Action: <input type="text"/>
4	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	Reagent: <input type="text"/>
5	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	Time: <input type="text"/>
6	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	
7	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	
8	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	
9	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	
10	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	

Procedure

Step 2

Yield: Density:

BP: - FP:

MP: - Vapor Pr:

Ballstien: Other Names:

FIG. 7

Synthematix Protocol Manager ver. 0.90b

Name: CAS: Formula: Weight:

Reagents: ☐ Enter procedure by hand

	CAS	Name	Weight	Amount (g)	
1	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	Action: <input type="text"/>
2	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	Qualifier: <input type="text"/>
3	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	Action: <input type="text"/>
4	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	Reagent: <input type="text"/>
5	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	Time: <input type="text"/>
6	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	
7	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	
8	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	
9	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	
10	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	

Procedure

Step 2

Qty.	<input type="text"/>	<input type="text"/>	Qty.	<input type="text"/>	<input type="text"/>
Qty.	<input type="text"/>	<input type="text"/>	Qty.	<input type="text"/>	<input type="text"/>
Qty.	<input type="text"/>	<input type="text"/>	Qty.	<input type="text"/>	<input type="text"/>
Qty.	<input type="text"/>	<input type="text"/>	Qty.	<input type="text"/>	<input type="text"/>
Qty.	<input type="text"/>	<input type="text"/>	Qty.	<input type="text"/>	<input type="text"/>

FIG. 8

Synthematrix Protocol Manager ver. 0.90b

Name: CAS: Formula: Weight:

Reagents: ☐ Enter procedure by hand.

	CAS	Name	Weight	Amount (g)	Action
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					

Search Results

- Class stir rod
- Overhead stirrer
- Teflon stir paddle

Procedure

Qty: stir

Qty:

Qty:

Qty:

Qty:

Qty:

Exit

FIG. 9

Synthematix Protocol Manager ver. 0.90q

File Apparatus Atmosphere Operations

Name: 6-(allylamino)-5-amino-4-chloropyrimidine CAS: R00-02-3 Formula: C7H9N4Cl Weight: 184.63

Reagents:

	CAS	Name	Weight	Amount (g)
1	R00-02-4	5-amino-4,6-dichloropyrimidine	163.99	50
2	107-11-9	2-Propen-1-amine	57.09	100
3	64-17-5	Ethanol	46.07	100
4	71-43-2	Benzene	78.11	3146.4
5				
6				
7				
8				
9				
10				

Starting Flask:

Equipped with:

Procedure

Step 1

Into a round bottom 3-neck flask was added <GN0> grams (<MN0> mol) of 5-amino-4,6-dichloropyrimidine

FIG. 10

Synthetix Protocol Manager ver. 0.90q

File Apparatus Atmosphere Operations

Name: 6-(allylamino)-5-amino-4-chloropyrimidine CAS: R00-02-3 Formula: C7H9N4Cl Weight: 184.63

Reagents: benzene ☐ Enter procedure by hand

	CAS	Name	Weight	Amount (g)
1	R00-02-4	5-amino-4,6-dichloropyrimidine	163.99	50
2	107-11-9	2-Propen-1-amine	57.09	100
3	64-17-5	Ethanol	46.07	100
4	71-43-2	Benzene	78.11	3146.4
5				
6				
7				
8				
9				
10				

Action:

Qualifier:

Action:

Reagent:

Time:

Procedure

Step 7

FIG. 11

Synthematrix Protocol Manager ver. 0.90q

File Apparatus Atmosphere Operations

Name: 6-(allylamino)-5-amino-4-chloropyrimidine CAS: R00-02-3 Formula: C7H9N4Cl Weight: 184.63

Reagents: benzene ☐ Enter procedure by hand

	CAS	Name	Weight	Amount (g)
1	R00-02-4	5-amino-4,6-dichloropyrimidine	163.99	50
2	107-11-9	2-Propen-1-amine	57.09	100
3	64-17-5	Ethanol	46.07	100
4	71-43-2	Benzene	78.11	3146.4
5				
6				
7				
8				
9				
10				

Action: To the flask
 Qualifier: was
 Action: added
 Reagent: Reagent 2
 Time:

Procedure

Step 2

To the flask was added <GN1> grams (<MN1> mol) of 2-Propen-1-amine and <GN2> grams (<MN2> mol) of Ethanol

FIG. 12

Synthetix Protocol Manager ver. 0.90q

File Apparatus Atmosphere Operations

Name: 6-(allylamino)-5-amino-4-chloropyrimidine CAS: R00-02-3 Formula: C7H9N4Cl Weight: 184.53

Reagents: benzene ☐ Enter procedure by hand

	CAS	Name	Weight	Amount (g)	
1	R00-02-4	5-amino-4,6-dichloropyrimidine	163.99	50	Action: <input type="text"/>
2	107-11-9	2-Propen-1-amine	57.09	100	Qualifier: <input type="text"/>
3	64-17-5	Ethanol	46.07	100	Action: <input type="text"/>
4	71-43-2	Benzene	78.11	3146.4	Reagent: <input type="text"/>
5					Time: <input type="text"/>
6					
7					
8					
9					
10					

Procedure

Step 2

To the flask was added <GN1> grams (<MN1> mol) of 2-Propen-1-amine and <GN2> grams (<MN2> mol) of Ethanol

FIG. 13

Synthematix Protocol Manager ver. 0.90b

Name: CAS: Formula: Weight:

Reagents: ☐ Enter procedure by hand

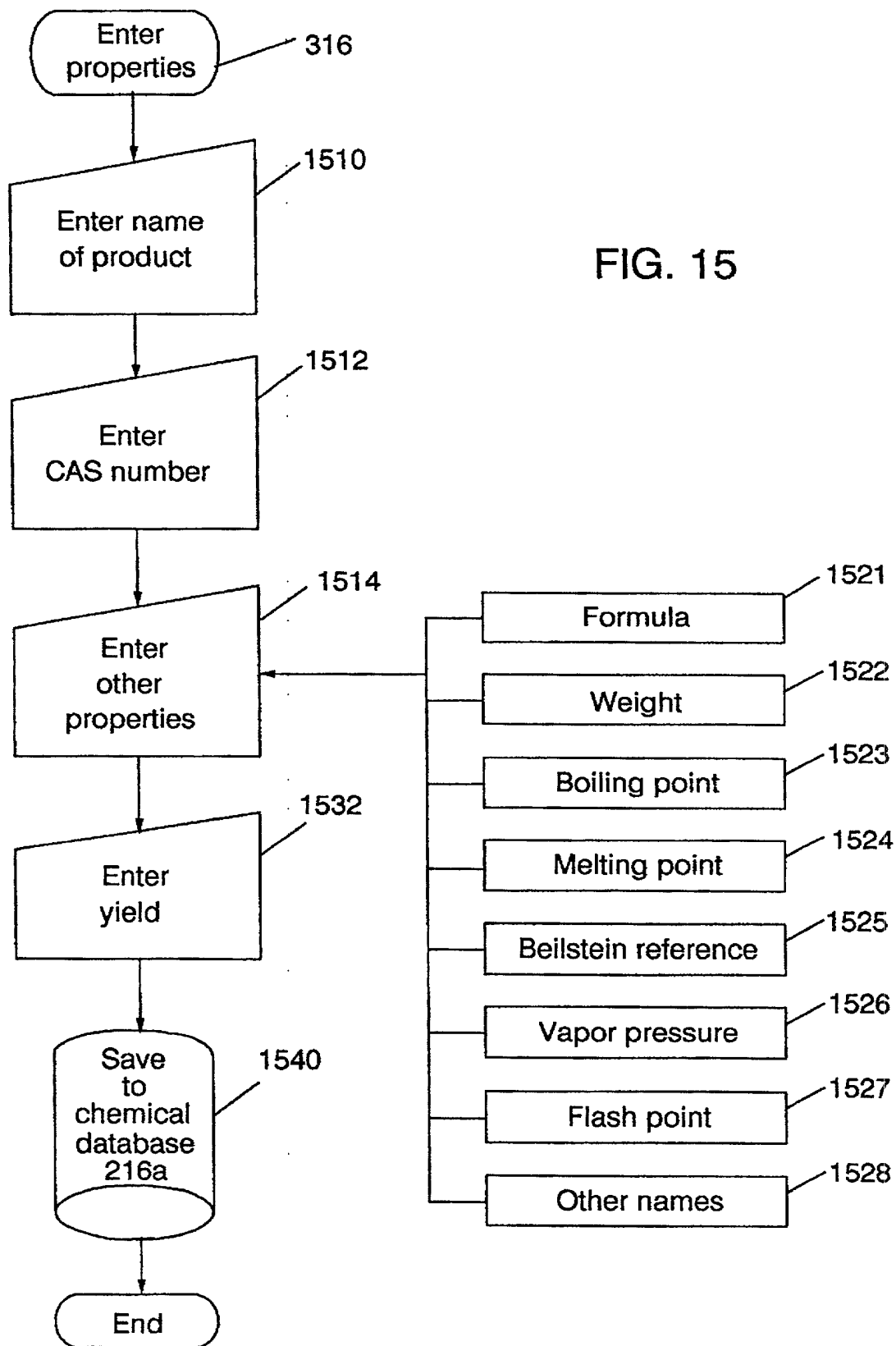
	CAS	Name	Weight	Amount (g)	
1	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	Action: <input type="text"/>
2	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	Qualifier: <input type="text"/>
3	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	Action: <input type="text"/>
4	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	Reagent: <input type="text"/>
5	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	Time: <input type="text"/>
6	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	
7	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	
8	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	
9	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	
10	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	

Procedure

Step 2

Reaction Type/Keywords (Separated by semicolon)

FIG. 14



File Apparatus Atmosphere Operations

Name: 6-(allylamino)-5-amino-4-chloropyrimidine CAS: R00-02-3 Formula: C7H9N4Cl Weight: 184.83

Reagents: benzene ☐ Enter procedure by hand

CAS	Name	Weight	Amount (g)	Action
R00-02-4	5-amino-4,6-dichloropyrimidine	183.99	50	
107-11-9	2-Propen-1-amine	57.09	100	

Author: Thomas et al. Journal: acid chemistry

Year: 1968 Vol: 1

Page: 22

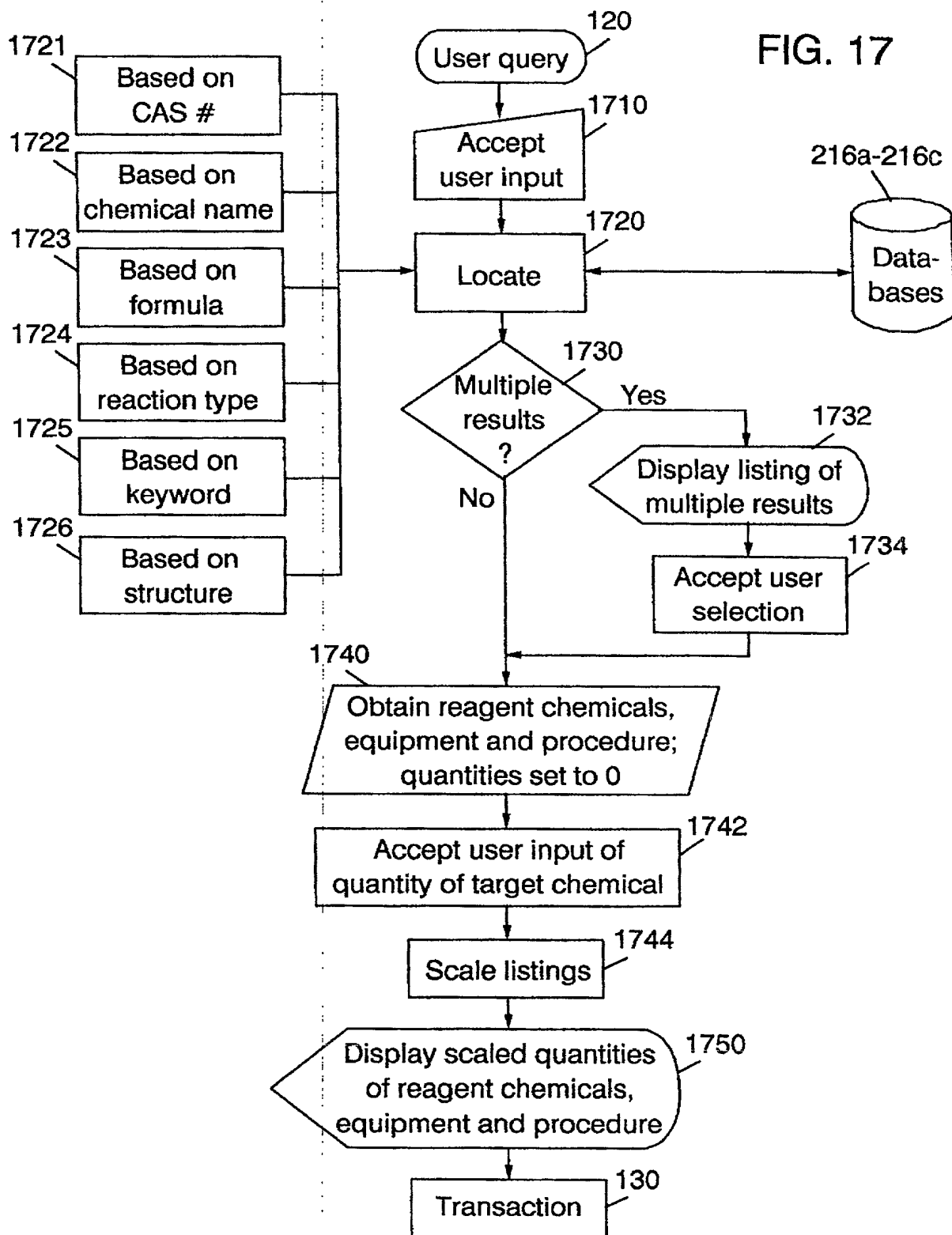
Procedure

Step 6

The extracts are then evaporated to dryness and can be recrystallized from petroleum ether to give a pure product

FIG. 16

FIG. 17



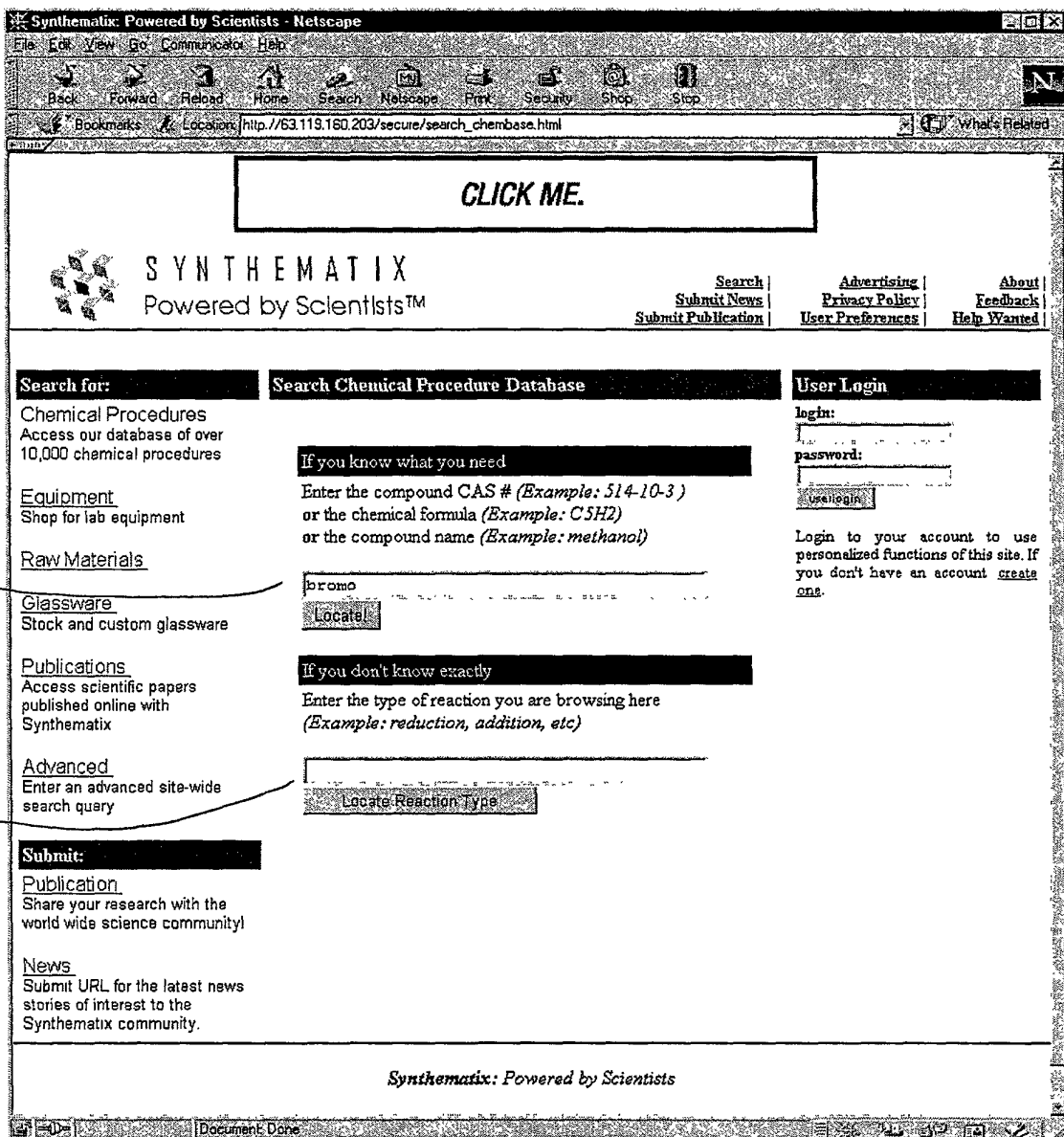



FIG. 18

Synthematix: Search Results - Netscape

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Bookmarks Location: <http://63.119.160.203/servlets/QueryProjectX?name=bromo> What's Related



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Synthematix: Search Results

Number of matches: 7

alpha-bromo-p-toluene boronic acid

n-Butyl-2-bromopropionate

(6-Bromo-2,2-diphenylbenzo[1,3]dioxol-5-yl)methanol

(6-Bromo-2,2-dimethylbenzo[1,3]dioxol-5-yl)methanol

6-bromo-2,2-diphenylbenzo[1,3]dioxole-5-carbaldehyde

6-bromo-3,4-[(diphenylmethylene)dioxy]-benzaldehyde ethanediyl acetal

1-Benzhydryl-3-bromoazetidine

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Document Done

FIG. 19

Synthetic Compound Procedure - Microsoft Internet Explorer

Address: <http://63.119.160.203/servlets/ShowChembase?id=16>

Links: [Home](#) [Science Resource](#) [Internet Start](#) [Windows Update](#) [RealPlayer](#) [HotBot](#) [Yahoo!](#)

n-Butyl 2-bromopropionate (209.08) C7H13BrO3

2-Bromopropionic Acid
C₃H₅BrO₂
Exact Mass 151.95
Mol Wt: 152.98
C: 23.55; H: 3.23;
Br: 52.23; O: 20.92

1-Butanol
C₄H₉O
Exact Mass 74.07
Mol Wt: 74.12
C: 64.82; H: 13.80; O: 21.59

n-Butyl 2-bromopropionate
C₇H₁₃BrO₃
Exact Mass 208.01
Mol Wt: 209.09
C: 40.21; H: 5.27; Br: 39.22; O: 15.30

Enter Mol Scale: mol

Chemicals needed

Chemical	Formula Weight	Equivalent	Moles Needed	Grams Needed
2-Bromopropionic acid	152.98	1	<input type="text" value="0.0000"/>	<input type="text" value="0.0000"/>
n-butanol	74.12	1	<input type="text" value="0.0000"/>	<input type="text" value="0.0000"/>
Hexane	86.18	1	<input type="text" value="0.0000"/>	<input type="text" value="0.0000"/>
Dowex 50W4-200	3.68	1	<input type="text" value="0.0000"/>	<input type="text" value="0.0000"/>
Water	18	1	<input type="text" value="0.0000"/>	<input type="text" value="0.0000"/>

Equipment needed

Qty	Equipment
1	Round bottom 3-neck flask
1	Overhead stirrer
1	Dean-Stark Trap
1	Condenser, Allihn, Dnp tip
1	Heating mantle
1	Recirculating Chiller
1	Heavy duty distillation head
1	Fraction Cutter
4	500 ml round bottom flask
2	Vanac
3	Tubing (ft.)

Procedure

- Into a 3 Neck Flask equipped with an overhead stirrer, Dean stark collector, condenser, and heating mantle was placed grams (mol) of 2-Bromopropionic acid
- To the 2-Bromopropionic acid was added grams (mol) of Butanol.
- To the 2-Bromopropionic acid/butanol mix was added grams (mol) of Hexane.
- To the flask is added grams of Dowex resin to facilitate the esterification. The resin is previously dried and has an activity of 3.68 grams/mol.
- Two vanacs are attached to the heating mantle and a setting of 50% power is used for heating to rapid reflux.
- The reaction is monitored for water generation. After collecting grams (mol) of water the reaction is complete.
- Hexane is then removed by continuous draining of the dean stark collector.
- The reaction is then cooled to room temperature and filtered through a fitted glass funnel to remove the Dowex resin.
- The crude n-butyl-2-bromopropionate is then purified via vacuum distillation. [The vacuum distillation procedure is below]

Vacuum distillation

- The product is placed into a 5L round bottom and connected to the pilot scale distillation head equipped with a vigreux column. The material is heated in a 5L mantle set to 45-50% power with a vanac.
- The product is collected at a head temp of 40-45°C. The material rapidly condenses once the apparatus is heated up. Total time for distillation is approx. 5 hours.

References

FIG. 20

2010

Synthetic: Compound Procedure - Microsoft Internet Explorer

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Back Forward Stop Refresh Home Search Favorites History Mail Print Edit Discuss Real.com

Address [?id=16&mol=12&MN0=12&GN0=1835.76&MN1=12&GN1=889.44&MN2=12&GN2=1034.16&MN3=12&GN3=44.16&MN4=12&GN4=216](#) Go

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2-Butyl-2-bromopropionate (209.08) C₇H₁₃BrO₃

2-Bromopropionic acid
C₃H₅BrO₂
Exact Mass: 151.95
Mol Wt: 152.98
C, 23.55; H, 3.29;
Br, 52.23, O, 20.92

1-Butanol
C₄H₁₀O
Exact Mass: 74.07
Mol Wt: 74.12
C, 64.82; H, 13.50; O, 21.59

n-Butyl 2-bromopropionate
C₇H₁₃BrO₂
Exact Mass: 208.01
Mol Wt: 209.08
C, 40.21; H, 6.27; Br, 38.22; O, 15.30

2060

2020

Enter Mol Scale: 12 mol Calculate

Chemicals needed

Chemical	Formula Weight	Equivalent	Moles Needed	Grams Needed
2-Bromopropionic acid	152.98	1	12.0000	1835.7600
n-butanol	74.12	1	12.0000	889.4401
Hexane	86.18	1	12.0000	1034.1600
Dowex 50W4-200	3.68	1	12.0000	44.1600
Water	18	1	12.0000	216.0000

2030

Equipment needed

Qty	Equipment
1	Round bottom 3-neck flask
1	Overhead stirrer
1	Dean Stark Trap
1	Condenser, Allihn, Drip tip
1	Heating mantle
1	Recirculating Chiller
1	Heavy duty distillation head
1	Fraction Cutter
4	500 ml round bottom flask
2	Variac
3	Tubing (ft.)

2040

Procedure

- Into a 3 Neck Flask equipped with an overhead stirrer, Dean stark collector, condensor, and heating mantle was placed 1835.7600 grams (12.0000 mol) of 2-Bromopropionic acid.
- To the 2-Bromopropionic acid was added 889.4401 grams (12.0000 mol) of Butanol.
- To the 2-Bromopropionic acid/butanol mix was added 1034.1600 grams (12.0000 mol) of Hexane.
- To the flask is added 44.1600 grams of Dowex resin to facilitate the esterification. The resin is previously dried and has an activity of 3.68 grams/mol.
- Two variacs are attached to the heating mantle and a setting of 50% power is used for heating to rapid reflux.
- The reaction is monitored for water generation. After collecting 216.0000 grams (12.0000 mol) of water the reaction is complete.
- Hexane is then removed by continuous drawing of the dean stark collector
- The reaction is then cooled to room temperature and filtered through a fritted glass funnel to remove the Dowex resin.
- The crude n-butyl-2-bromopropionate is then purified via vacuum distillation. [The vacuum distillation procedure is below]

Vacuum distillation

- The product is placed into a 5L round bottom and connected to the pilot scale distillation head equipped with a vigreux column. The material is heated in a 5L mantle set to 45-50% power with a variac
- The product is collected at a head temp of 40-45°C. The material rapidly condenses once the apparatus is heated up. Total time for distillation is approx. 5 hours.

2050

References

FIG. 21

FIG. 22

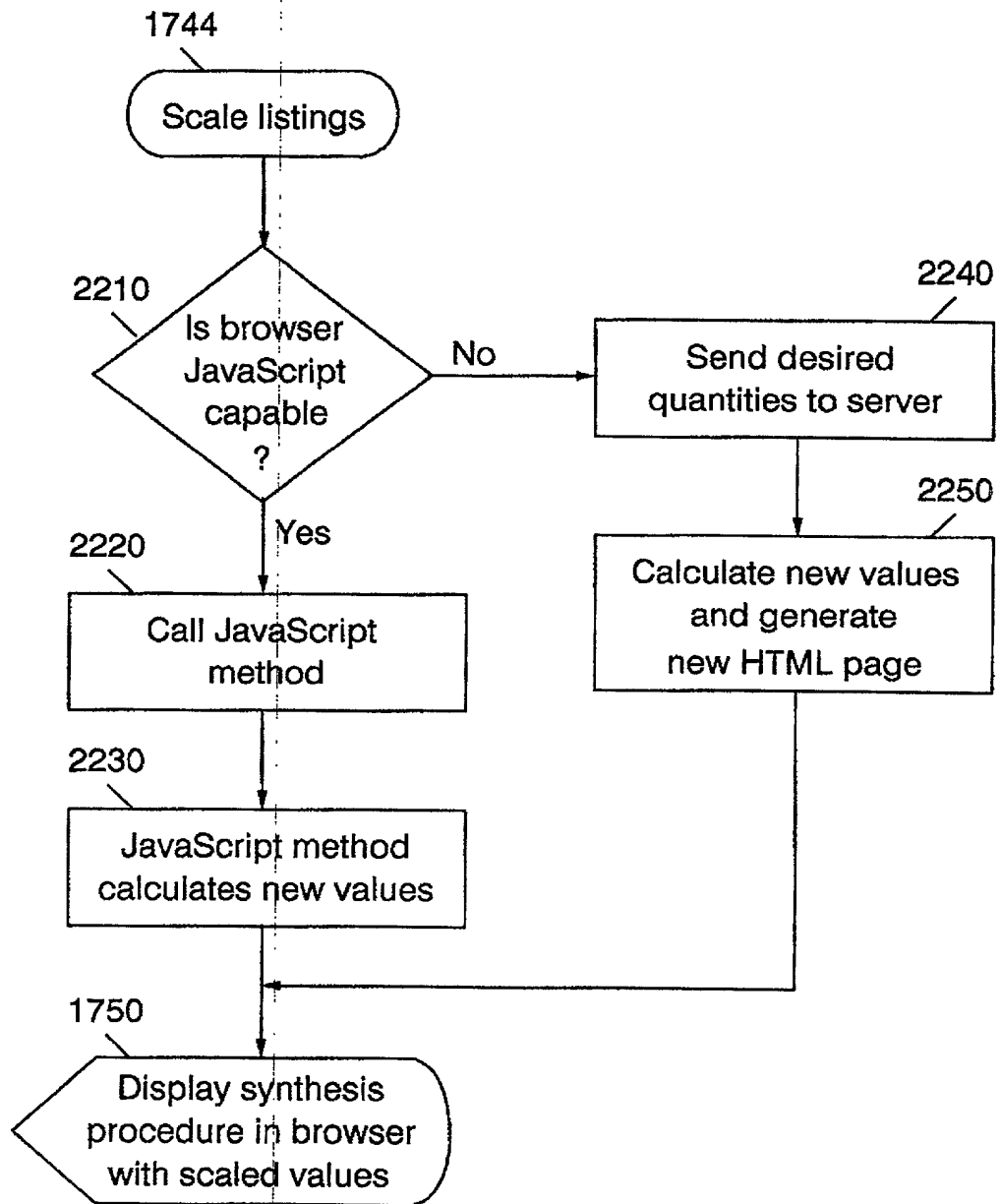


FIG. 23

